

# Ion Plated Gold Films: Properties, Tribological Behavior and Performance

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## ION PLATED GOLD FILMS: PROPERTIES, TRIBOLOGICAL BEHAVIOR AND PERFORMANCE

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Abstract

The glow discharge energizing favorably modifies and controls the coating/substrate adherence and the nucleation and growth sequence of ion plated gold films. As a result the adherence, coherence, internal stresses, and morphology of the films are significantly improved. Gold ion plated films because of their graded coating/substrate interface and fine uniform densely packed microstructure not only improve the tribological properties but also induce a surface strengthening effect which improves the mechanical properties such as yield, tensile, and fatigue strengths. Consequently significant improvements in the tribological performance of ion plated gold films as compared to vapor deposited gold films are shown in terms of decreased friction/wear and prolonged endurance life.

Introduction

Ion plating is an ion assisted or glow discharge deposition technique which offers great flexibility in tailoring the coating-surface properties which are independent of the bulk properties. It is well established that the properties of the surface-subsurface influence the performance of materials which are used in technological applications through processes such as friction, wear, corrosion, erosion, fatigue, electrical, and thermal contacts which are all basically surface initiated and lead to failures.

Thin ion plated soft metallic lubricating films such as gold, silver, and lead have shown major improvements in tribological performance over films deposited by the other deposition techniques. Ion plated gold films reduce friction and/or wear on sliding or rotating surfaces, and are finding increased applications in areas where combination of high vacuum, high temperature, and radiation preclude the use of conventional lubricants such as oils and greases. These soft films confine friction losses to a thin, low shear strength film interposed between the contacting surfaces. Gold ion plated films are widely used in spaceborn bearings of various satellite mechanisms such as solar drives, de-spin assemblies and gimbals or under adverse environmental conditions of high vacuum, high temperature, and radiation. These ion plated films display reduced coefficient of friction, reduced wear and an extended endurance life, and also alter the mode of debris generation and reduce torque noise.

Basically, thin, soft metallic film lubrication is effective for wear reduction when adhesive wear predominates, but is of little value in abrasive wear, therefore the most important requirement is strong adherence. Ion plated metallic films have exceptionally strong adherence which is attributed to a graded film/substrate interface, regardless of film/substrate compatibility (solid solubility).

The objective of this paper is to discuss and characterize the unique properties of ion plated gold films in terms of adhesion, cohesion, microstructure, internal stresses, morphology, film thickness, and hardness as it affects the tribological performance through effective lubrication.

Ion Plating Process

Ion plating is an ion assisted or glow discharge deposition technique, in which ions or energetic atoms transfer energy, momentum, and charge to the substrate and where the film grows in a manner which can be controlled favorably to modify surface, subsurface chemistry and microstructure. During ion plating the energizing is provided by the gas discharge and in principle it combines (1) the energetic impingement of ions and activated atoms of the sputtering process, (2) the high throwing power of electroplating and (3) the high deposition rates of thermal evaporation. The impingement of the ions and neutrals contributes to the excellent adherence and modified structural growth of the film. The high throwing power provides for three-dimensional coverage to coat complex, intricate surfaces. The basic ion plating system consists of a dc-diode configuration, where the specimen to be coated is made the cathode of the high voltage dc-circuit and the evaporation source the anode. This is shown schematically and photographically in Fig. 1. The principles and operation of the process have been widely described in the literature [1-6] and will not be reviewed here.

Typical used commercial ion plating conditions are: voltage 3 to 5 kV, argon pressure 20 mtorr and cathode current density 0.3 to 0.5 mA/cm<sup>2</sup>. It has been estimated that under these conditions the ions carry only 10 percent of the energy dissipated while the energetic neutrals carry 90 percent [2]. The energetic neutrals which are generated through charge transfer collisions (where a large part of the kinetic energy of the ions are transferred to the neutral atoms) constitute a very significant proportion of the energy carried. The ions and the activated neutrals may have a distribution of energies from thermal 0.2 eV up to the voltage applied to the discharge. It has been estimated that the average energies of the ions and neutrals are of the order of 100 eV [2]. This glow discharge energizing during ion plating favorably modifies the nucleation and growth sequence and the coating/substrate adherence. As a result, adherence, coherence, density, internal stresses, morphology, and defects in the coatings can be significantly improved, which in turn favorably affect the lubricating properties.

Adherence and Graded Interface

Strong adherence between the coating and the substrate is of paramount importance for any successful application. The interface or transition from the substrate material to the coating usually forms a break in the normally uniform crystallinity and/or composition. This abrupt disparity or mismatch is reflected in the coefficient of thermal expansion, thermal conductivity, and hardness. The objective is to reduce or eliminate this abrupt disparity by inducing a graded or fused interface between the coating and the substrate. The process of ion plating provides a gradual composition or graded interface. This effect is illustrated with experimental results by analyzing ion plated gold films on nickel and iron surfaces by x-ray photoelectron spectroscopy (XPS) depth profiling as shown in Figs. 2(a) and (b) [7,8]. Compositions of surface (at %) for gold films ion-plated on nickel

and iron were obtained as functions of the sputtering removal time from XPS analysis. The composition of gold did not change in the first 20 min of sputtering and thereafter gradually decreased with increasing sputtering time. However, the nickel and iron content increased with increased sputtering times. The graded interface for gold on nickel was approximately 6  $\mu\text{m}$  thick; that for gold on iron was approximately 1.5  $\mu\text{m}$  thick. The deeper graded interface between gold and nickel is due to the continuous solid solubility between the two metals. Since gold and iron have very limited solid solubility (1.5 at % at 850 °C) the interface is reduced and is basically produced by implantation and ion-atomic mixing. On the other hand, XPS depth profiles for gold vacuum deposited on nickel and steel surfaces are shown in Fig. 3, where the composition of gold rapidly decreased with increasing sputtering time, thus forming an abrupt interface.

The interface formation was also investigated by Micro-Vickers hardness measurements where indentation hardness depth profiles were established for the ion plated and vacuum deposited gold films on nickel substrates and are shown in Figs. 4(a) and (b). The microhardness of the ion plated gold film, graded gold-nickel interface and nickel substrate as a function of depth from the surface is shown in Fig. 4(a). The gold films were gradually removed by argon ion sputtering for microhardness measurements. Initially, the microhardness of the gold film is relatively low, but gradually increases in the interface region and finally decreases again as it reaches the nickel substrate. The higher hardness in the interface region is due to alloying effects. The vapor deposited gold film on nickel substrates shown in Fig. 4(b) exhibits a constant hardness, which indicated the lack of alloying.

The XPS and microhardness depth profiles experimentally verify the formation of the gold graded-fused interface which is responsible for the excellent adherence. The gold graded interface not only provides excellent adherence, but also induces a surface strengthening effect which improves the mechanical properties such as yield strength, tensile strength, and fatigue strength as shown in Figs. 5(a) and (b). The surface-subsurface strengthening may arise from solid-solution alloying, dispersion of mobile interstitial or substitutional atoms which act as barriers to the egress of dislocations.

#### Nucleation and Growth Morphology

During ion plating the evaporant flux of energetic ions and atoms transfers energy, momentum, and charge to the substrate and the depositing film surface. Because of the low ionization efficiency of the dc-diode process, gold atoms are expected to arrive at the substrate with a range of energies. The high energy gold species arrive at the substrate with energies high enough to cause particle implantation into the substrate where they lose their energy, rather than diffuse over the surface. Continuous ion bombardment of the substrate during ion plating sputters off the loosely bonded atoms, thus leaving a state of high energy implanted particles which act as nucleation sites. Ion plated gold films unlike the conventional films exhibit a distinct nucleation behavior as shown in Figs. 6(a) to (c). The nuclei formed during ion plating exhibit these distinct characteristics. The size is considerably smaller (150 Å) and has a high nuclei density with a uniform distribution. As deposition continues the nuclei remain rounded with only a slight increase in size, less than 20 percent, without the typical island coalescence which is typical during conventional nucleation. In classical nucleation, there is a decreasing tendency for them to become completely round after coalescence, consequently islands become elongated and join to form an irregular network

structure. As deposition continues in ion plating, the existing nuclei grow, but formation of new nuclei continues to be a major growth mechanism [9]. Consequently, continuous films are formed in the 250 Å thickness range, with fine uniform grain structure, high packing density, and a minimum lattice misfit. A typical ion plated gold film as compared to a vapor deposited one is shown in Fig. 7, where a high degree of nonuniformity in grain size and shape is obvious for the vapor deposited film.

#### Film Thickness Effects

In thin film lubrication the thickness of gold films has a very pronounced effect on the coefficient of friction as shown in Fig. 8 with ion plated Au films. The effective or minimum film thickness for gold films was about 2000 to 3000 Å with the minimum coefficient of friction of 0.1 [10].

Based on the coefficient of friction, the variation of film thickness can be divided into two regions as shown in Fig. 9. The ultra thin and the thin film region with an effective or critical film thickness at which the friction coefficient reaches a minimum value. Film thickness lower than that of minimum friction did not provide full surface coverage, and some steel to steel contact occurs. If film thickness is higher than the minimum friction, an increasing proportion of the load capacity was carried by the metallic film, thus progressively reducing the effect of the hard substrate. By reducing the thickness, the junction shearing strength is determined by the soft metallic film, while the contact surface and the yield point are determined by the properties of the support material. The relationship between the critical film thickness and the minimum friction coefficient is affected by the surface roughness and the deposition technique selected. With rough surfaces the friction coefficient increases continuously as the film thickness increases without going through a minimum [11,12].

#### Internal Stresses

The total internal stress is composed of the thermal stress  $\sigma_t$  and intrinsic stress  $\sigma_i$ , where  $\sigma_{tot} = \sigma_t + \sigma_i$ . The thermal stress is due to the differences in the thermal expansion coefficient of the coating and the substrate material. Intrinsic stress is due to the accumulating effects of crystallographic defects or flows formed into the coating during deposition. Usually, the intrinsic stresses in the film are the dominant part of the total stress since they are microstructure-sensitive. During ion plating the presence of gas discharge and primarily the high energy of the incident atoms and ions increases the substrate temperature and favors the densification of the microstructure. As a result gold ion plated films generally show little or no internal stresses (approximately  $5 \times 10^7$  dyn/cm<sup>2</sup>).

It should be also pointed out, that ion plated films in contrast to evaporated ones generally show compressive stresses instead of tensile. This should not be surprising, considering their dense microstructure.

#### Frictional Characteristics

Ion plated gold films because of their excellent adherence and dense film microstructure have found extensive uses in spaceborn bearings of satellite mechanisms. Typical friction curves for ion plated and vapor deposited gold films 2000 Å thick as determined in a pin and disk tribotester under vacuum conditions are shown in Fig. 10. The ion plated Au films had three distinct improvements over the vapor deposited ones: (1) increased endurance life, (2) lower

coefficient of friction and (3) avoidance of catastrophic failure. The reasons for the increased endurance life are attributed to the superior adherence, the lower coefficient of friction due to the dense, cohesive small crystalline size and the optimum film thickness, and the gradual increase in the coefficient of friction after the film has been worn off to the formation of the graded interface. The graded gold interface formed during ion plating is of dual importance in gold film lubrication, since a graded interface which is generated is responsible for the excellent adherence and prevention of unexpected seizure at tribocontacts.

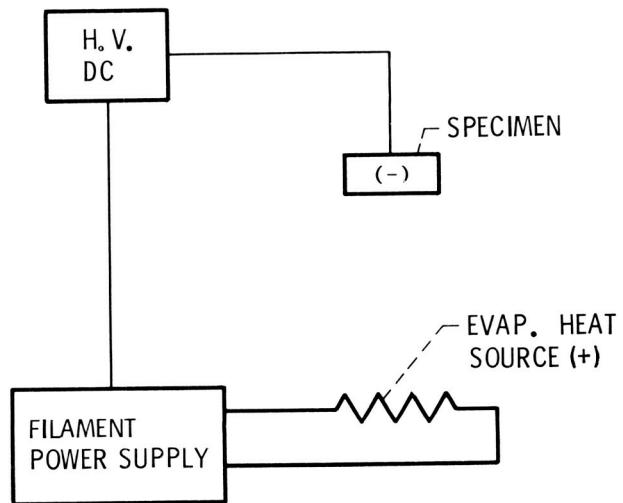
### Conclusions

During ion plating the glow discharge energizing favorably modifies and controls the coating/substrate adherence and the nucleation and growth sequence. The graded interface formed not only guarantees an excellent adherence, but also induces a surface strengthening effect which improves the mechanical properties. The ion plated gold films 0.2  $\mu\text{m}$  thick exhibit a distinct, improved film structure with an equiaxed, small grain size which is responsible for the high packing density and a minimum amount of lattice misfit. As a result, continuous, uniform films are obtained at lower nominal thicknesses which favorably affects the coefficient of friction. The strong adherence with the graded interface and the dense microstructure improves the tribological performance of sliding or rotating surfaces in terms of a lower coefficient of friction, lower wear, increased endurance lives and avoidance of catastrophic failure after lubricating film depletion.

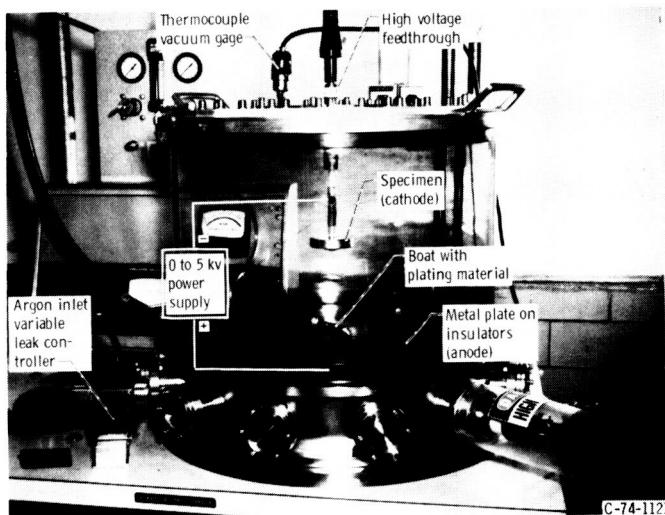
### REFERENCES

1. D.M. Mattox, "Ion Plating Technology," in Deposition Technologies for Films and Coatings, R.F. Bunshah, ed., Park Ridge, IL: Noyes Publications, 1982, pp. 244-287.
2. D.G. Teer, "Adhesion of Ion Plated Films and Energies of Deposition," J. Adhes., vol. 8, pp. 289-300, 1977.
3. D.M. Mattox, "Fundamentals of Ion Plating," J. Vac. Sci. Technol., vol. 10, pp. 47-52, 1973.
4. D.M. Mattox, Sputter Deposition and Ion Plating Technology, New York: American Vacuum Society, 1973.
5. D.M. Mattox, "Mechanisms of Ion Plating," in Proceedings of the International Conference on Ion Plating and Allied Techniques, (IPAT 79), CEP Consultants Ltd., Edinburgh, England, pp. 1-10, 1979.
6. T. Spalvins, "Survey of Ion Plating Sources," J. Vac. Sci. Technol., vol. 17, pp. 315-321, 1980.
7. K. Miyoshi, T. Spalvins, and D.H. Buckley, "The X-Ray Photoelectron Spectroscopy Depth Profiling and Tribological Characterization of Ion-Plated Gold on Various Metals," Thin Solid Films, vol. 108, pp. 199-207, 1983.
8. K. Miyoshi, T. Spalvins, and D.H. Buckley, "Tribological Characteristics of Gold Films Deposited on Metals by Ion Plating and Vapor Deposition," in 3rd International Conference on Solid Lubrication, ASLE SP-14, Park Ridge, IL: ASLE, pp. 208-216, 1984.
9. G.S.A. Shawki, M.G. El-Sherbiny, and F.B. Salem, "Nucleation and Interface Formation in Thin Films," Thin Solid Films, vol. 75, pp. 29-36, 1981.
10. T. Spalvins and B. Buzek, "Frictional and Morphological Characteristics of Ion-Plated Soft Metallic Films," Thin Solid Films, vol. 84, pp. 267-272, 1981.
11. R.D. Arnell and F.A. Soliman, "The Effects of Speed, Film Thickness and Substrate Surface Roughness on the Friction and Wear of Soft Metal Films in Ultrahigh Vacuum," Thin Solid Films vol. 53, pp. 333-347, 1978.
12. M.A. Sherbiny and J. Halling, "Friction and Wear of Ion-Plated Soft Metallic Films," Wear, Vol. 45, pp. 211-220, 1977.

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(a) Schematic.



(b) Ion plating chamber.

Figure 1. - Ion plating system.

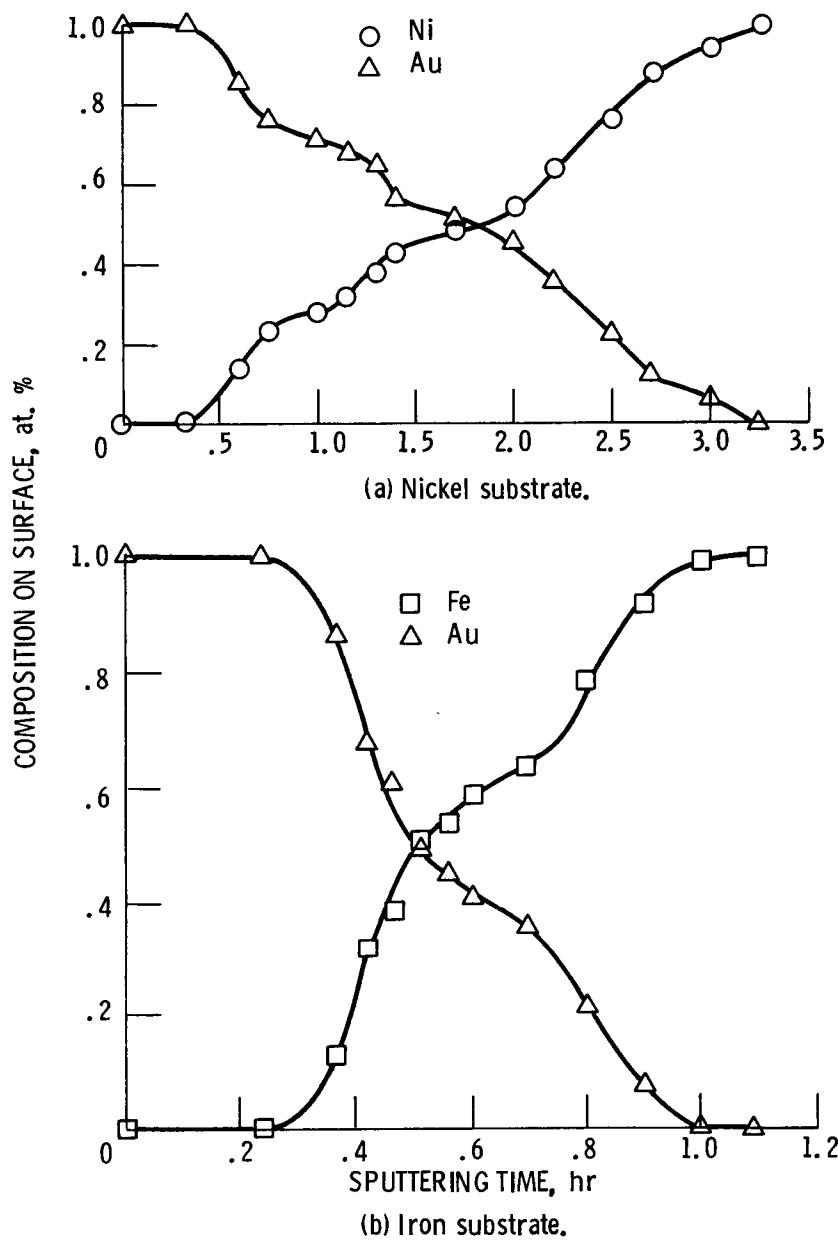
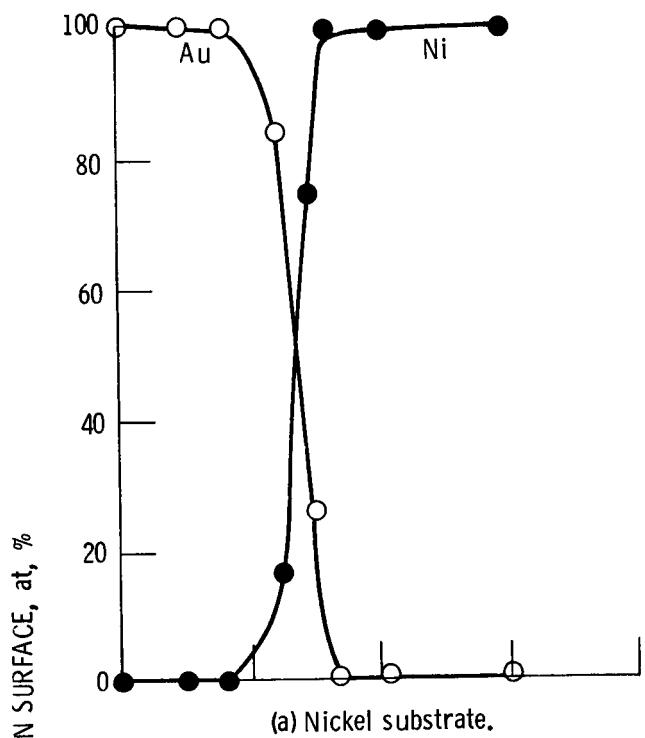
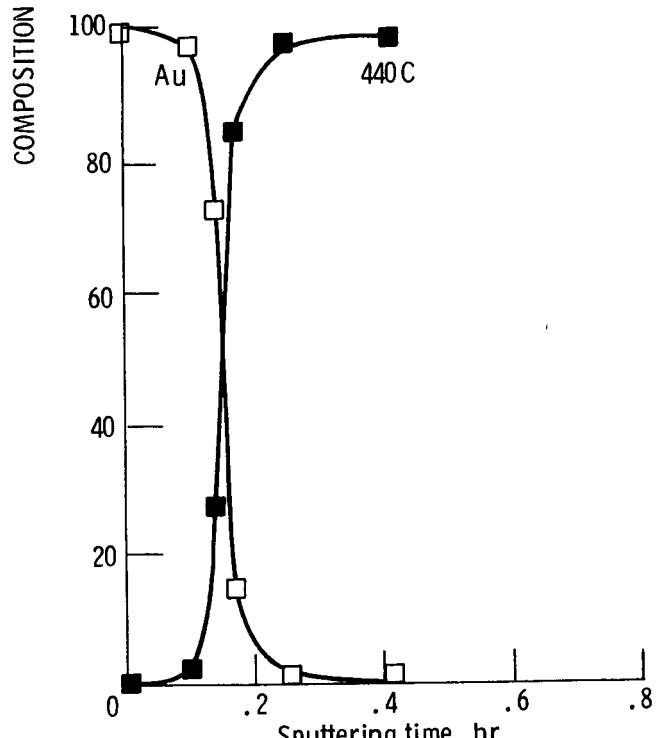


Figure 2. - Elemental depth profiles for gold ion plated on nickel and iron. Film thickness,  $0.6\mu\text{m}$ .



(a) Nickel substrate.



(b) 440C substrate.

Figure 3. - Element depth profiles for gold vapor deposited on copper, nickel, and 440C stainless steel. Film thickness, 0.3  $\mu$ m.

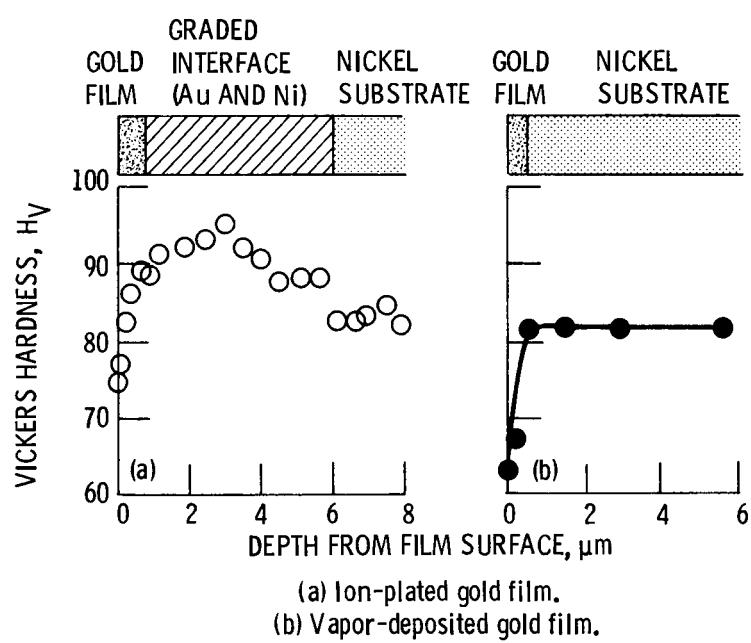
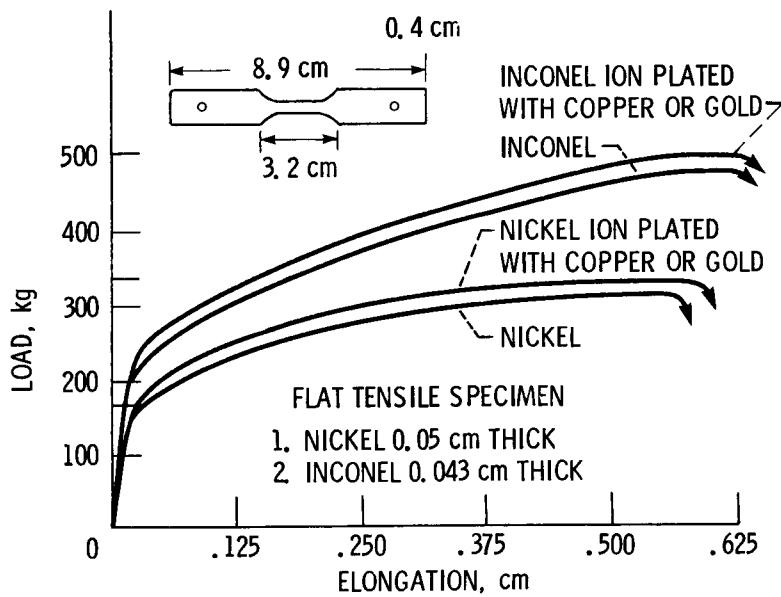
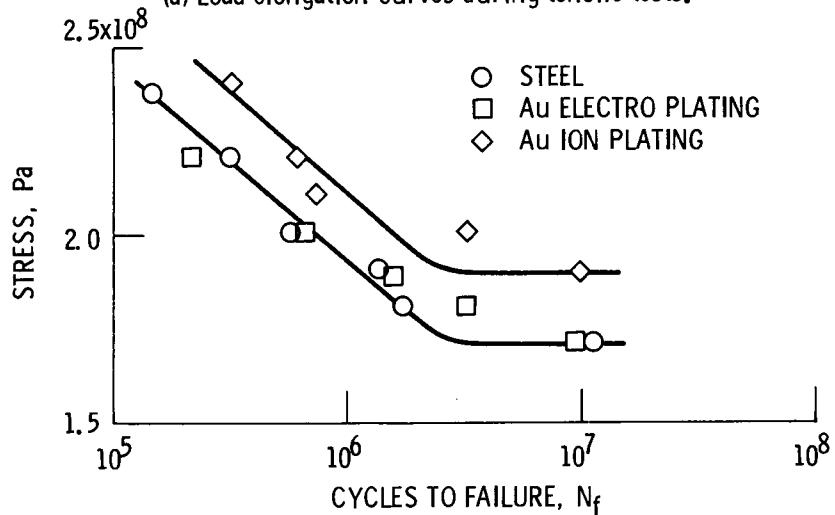


Figure 4. - Hardness depth profiles for gold ion plated and vapor deposited on nickel substrate. Hardness measuring load, 0.1 N.

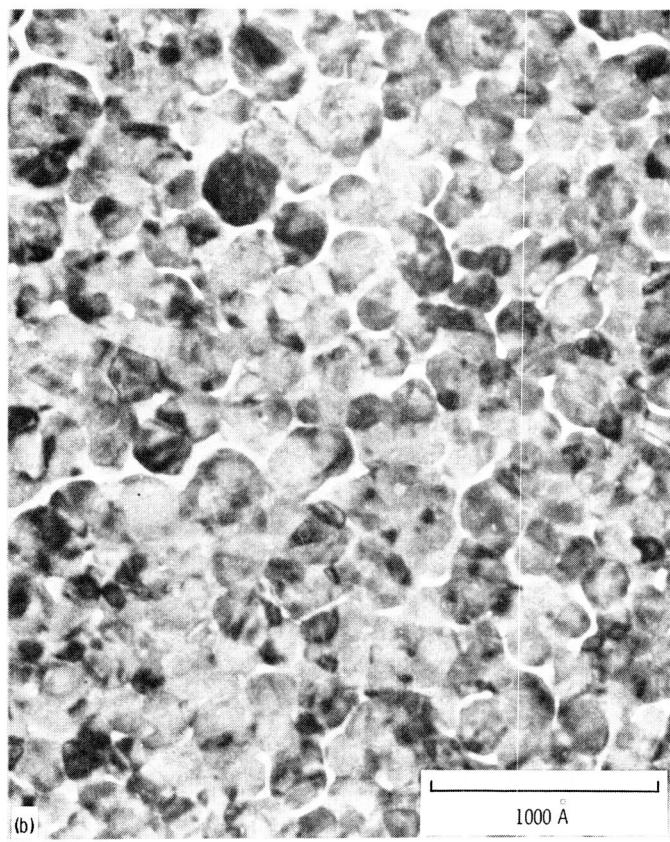
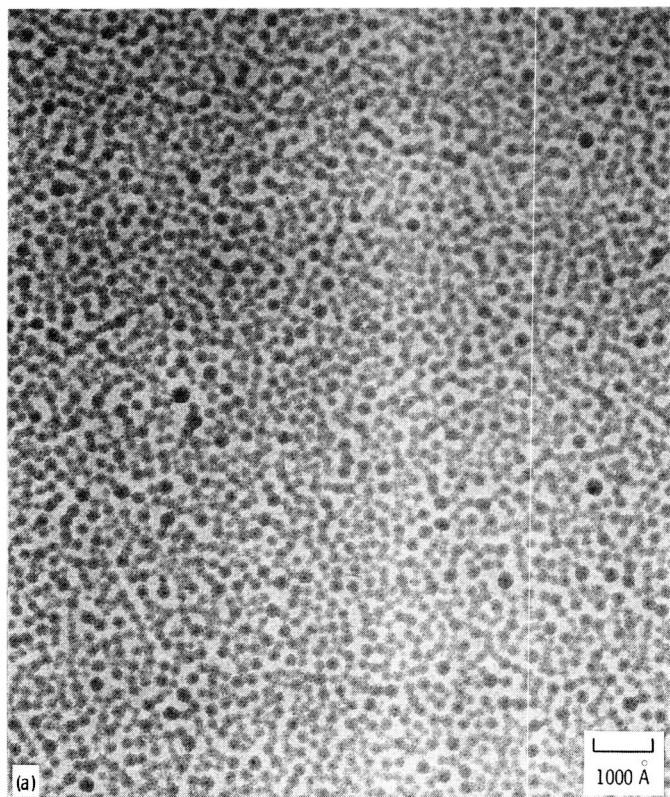


(a) Load elongation curves during tensile tests.



(b) Effect of ion plating on fatigue property of low carbon steel.

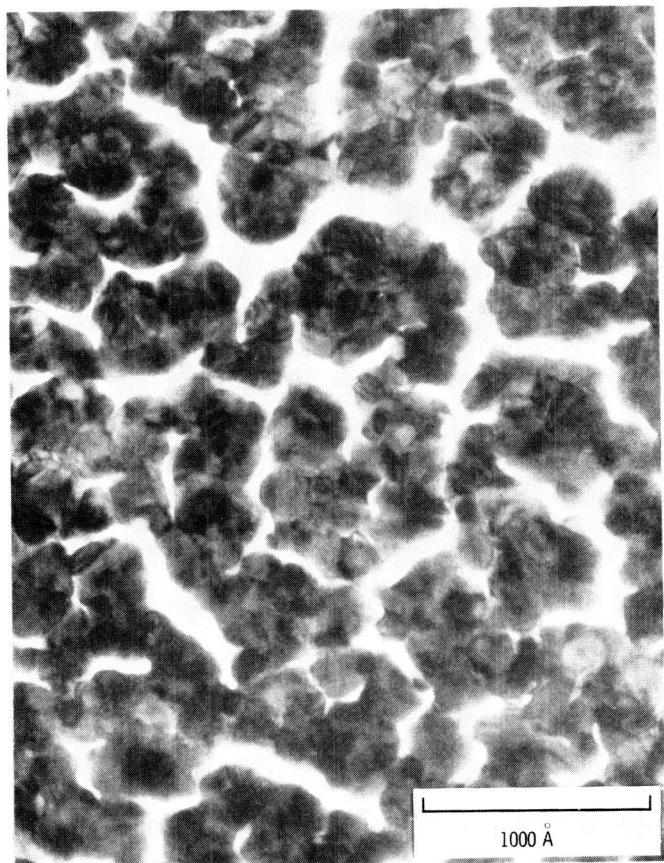
Figure 5. - Surface strengthening effects on mechanical properties.



ION PLATED

Figure 6. - Transmission electron micrographs<sup>1</sup> of gold during nucleation.

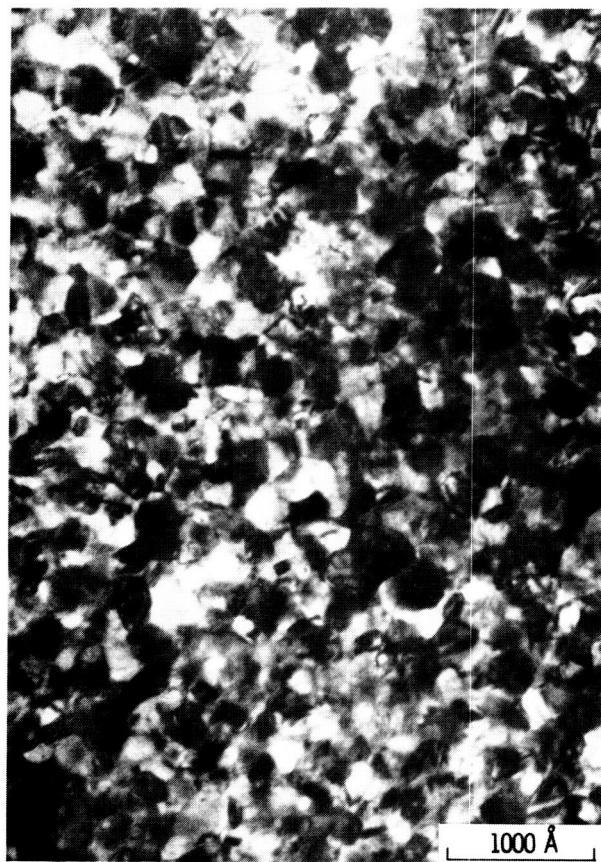
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Figure 6. - Concluded.

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(a) ION PLATED GOLD.



(b) VAPOR DEPOSITED GOLD.

Figure 7. - TEM micrograph of the crystal structure of a gold film.

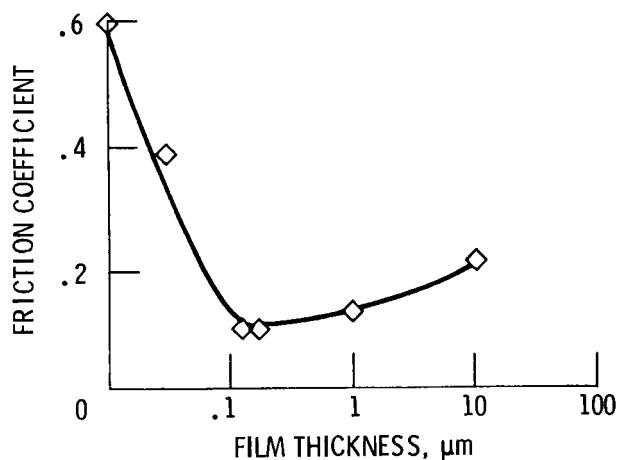


Figure 8. - The variation of friction coefficient with gold film thickness (load 2.45N; speed  $1.52 \text{ m min}^{-1}$ ; pressure  $2 \times 10^{-5} \text{ torr}$ ; roughness  $0.02 \mu\text{m}$ ).

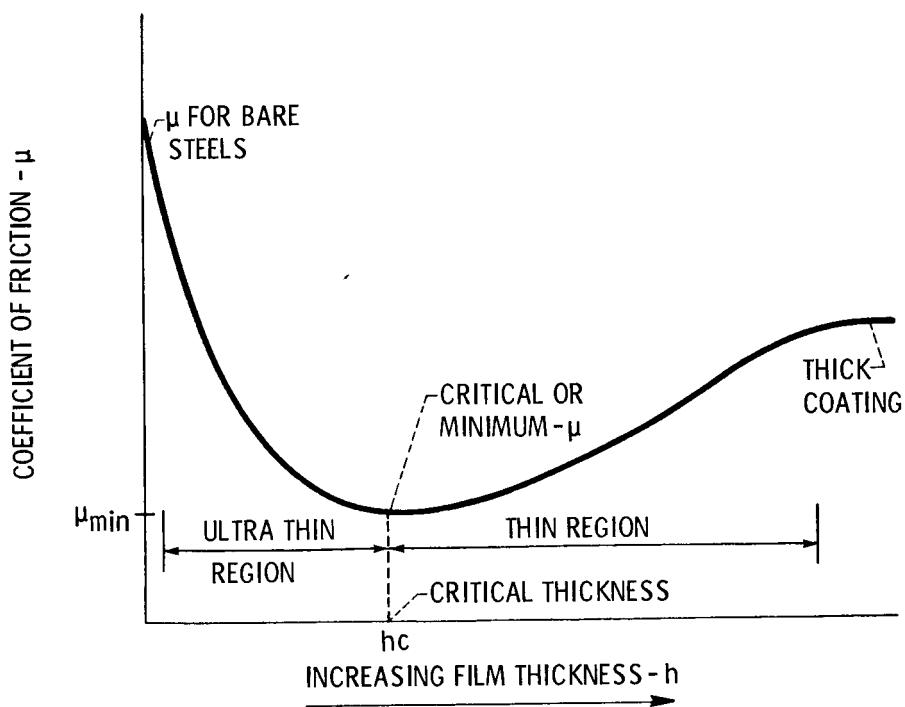


Figure 9. - Schematic representation of the effect of film thickness on the friction coefficient of low shear strength metallic films on steel.

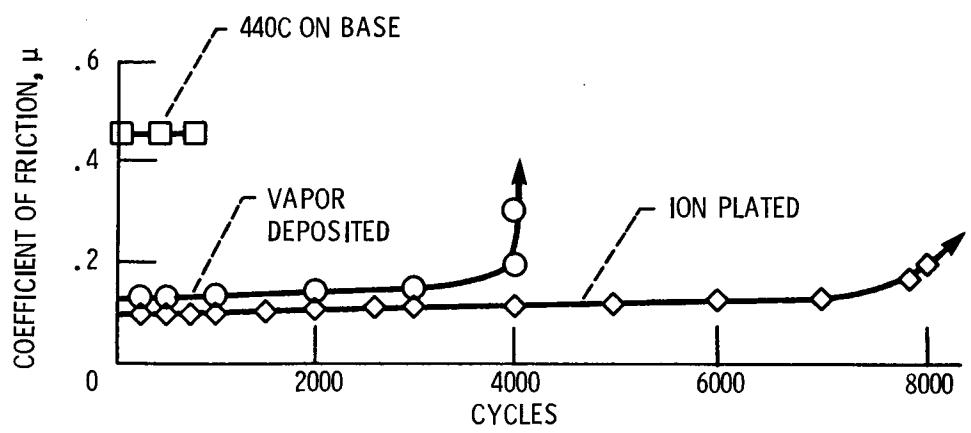


Figure 10. - Comparison of coefficient of friction of ion plated and vapor deposited gold film on 440C steel.

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